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environmental restoration project

A Department of Energy
Environmental Cleanup Program

Environmental Restoration Project
Standard Operating Procedure

for:

Routine Validation of Inorganic Data

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Routine Validation of Inorganic Data

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List of Acronyms and Abbreviations

CCB	continuing calibration blank
CCV	continuing calibration verification
CLP	Contract Laboratory Program
COC	chain of custody
CRDL	contract-required detection limit
EDL	estimated detection limit
EPA	US Environmental Protection Agency
ER	environmental restoration
GFAA	graphite furnace atomic absorption
ICB	initial calibration blank
ICPES	inductively coupled plasma emission spectroscopy
ICS	interference check sample
ICV	initial calibration verification
IDL	instrument detection limit
LANL	Los Alamos National Laboratory
LCS	laboratory control sample
MDL	method detection limit
n/a	not applicable
%R	percent recovery
RPD	relative percent difference
SMO	Sample Management Office
SOP	standard operating procedure
SOW	statement of work

Routine Validation of Inorganic Data

NOTE: Environmental Restoration (ER) Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/documents/Procedures/sops.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear.

1.0 PURPOSE

This standard operating procedure (SOP) represents the minimum standard for evaluating routine inorganic analytical data. These data can be generated for the Los Alamos National Laboratory (LANL) ER Project using SW-846 Method 6010, 6020, or 7000 series or the comparable Contract Laboratory Program (CLP) methods under the current statement of work (SOW) for analytical services (LANL 1995). The evaluation of data by this procedure is not specific to a particular data use, although this procedure may be used as a point of departure for developing focused data validation requirements specific to a particular data use.

Note: Implementation of this procedure results in a tabulation of data compliances and noncompliances identified relative to expectations based on national guidelines for data review (EPA 1994). Because the acceptance criteria used for this procedure are not based on site-specific acceptance criteria, the results of this validation procedure are intended to be used as *general indicators* of data quality and should not be construed as a definitive identification of data usability.

Note: Implementation of this procedure may be followed by a more focussed and data-use-specific evaluation of the data, especially if implementation of this SOP indicates that the data may contain technical deficiencies.

2.0 TRAINING

All data validators implementing this SOP shall possess a minimum of a bachelors degree in chemistry and two years experience in generating analytical data in an environmental analytical laboratory or two years of data-validation experience. New validators shall work under the direct supervision of an experienced ER Project validator. The work of new validators shall be reviewed and signed by an experienced ER Project validator until ten data record packages for each analytical suite have been satisfactorily validated. ER Project validators shall have demonstrated familiarity with the US Environmental Protection Agency (EPA) national functional guidelines for data review. All data validators must document

that they have read and understand this SOP and completed all applicable training assignments in accordance with QP-2.2.

3.0 DEFINITIONS

3.1 *Continuing calibration verification (CCV)* — Combination of calibration blank and check standards used to determine if the instrument response to analyte concentration is within acceptable bounds relative to the initial calibration. A continuing calibration is performed every 12 hrs of operation and establishes the 12-hr relative response factors on which quantitations are based, thus verifying the satisfactory performance of an instrument on a day-to-day basis.

3.2 *Data validator* — A person who has met the minimum standards of training established in Section 2.0 and who implements this SOP on behalf of the ER Project.

3.3 *Detect* — A sample result above the method detection limit (MDL) as reported by the contract analytical laboratory. The contract laboratory reports the concentration of the analyte in the sample.

3.4 *Duplicate analysis* — Analysis performed on one of a pair of identically prepared subsamples taken from the same sample.

3.5 *Duplicate measurement* — An additional measurement performed on a prepared sample under identical conditions to evaluate the variance in the measurement.

3.6 *Estimated detection limit (EDL)* — The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine analytical-laboratory operating conditions. The low point on a calibration curve should reflect this quantitation limit. The EDL is not used to establish detection status. For the ER Project, the EDL reflects the contract-required detection limits (CRDLs) of the EPA Contract-Laboratory Program (CLP) methods.

3.7 *Holding time* — The maximum elapsed time from sample collection to sample preparation and/or analysis that a sample can be stored without unacceptable changes in analyte concentrations. Holding times apply under prescribed storage conditions; deviations in storage conditions may affect holding times. Appropriate storage conditions for samples of various matrices scheduled for selected analyses may be found in the current LANL-ER-SOP-1.02, the applicable analytical method, and the current ER Project SOW for analytical services.

3.8 *Initial calibration* — Process used to establish the relationship between instrument response and analyte concentration at several analyte-

concentration values to demonstrate that an instrument is capable of acceptable analytical performance.

3.9 *Instrument detection limit* — The IDL is defined to be three times the average of the standard deviations obtained on three nonconsecutive days from the *analysis* of a standard solution, with seven consecutive measurements of that solution per day. The standard solution must be prepared at a concentration of three to five times the instrument manufacturer's estimated IDL. This is a measure of instrument sensitivity without any consideration for contributions to signal from reagents.

3.10 *Interference-check sample (ICS)* — A sample used to verify the contract analytical laboratory's interelement and background correction factors for inductively coupled plasma emission spectroscopy (ICPES) analyses. The ICS shall be analyzed a minimum of twice in each eight-hour shift or at the beginning and end of each analysis run, whichever is more frequent.

3.11 *Laboratory control sample (LCS)* — A known matrix that has been spiked with compound(s) representative of the target analytes. The LCS is used to document laboratory performance. The acceptance criteria for LCSs are method specific.

3.12 *Laboratory duplicate sample* — The portions of a sample taken from the same sample container, prepared for analysis and analyzed independently but under identical conditions; used to assess or demonstrate acceptable laboratory method precision at the time of analysis. Each duplicate sample is expected to be equally representative of the original material. Duplicate analyses also are performed to generate data, to determine the long-term precision of an analytical method on various matrices.

3.13 *Laboratory qualifier (or laboratory flag)* — Codes applied to the data by the contract analytical laboratory to indicate, on a gross scale, a verifiable or potential data deficiency. These flags are applied using the EPA CLP guidelines.

3.14 *LANL data validation qualifiers* — The data qualifiers defined by LANL and used in the ER Project baseline-validation process. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate ER Project SOP (ER-SOPs 15.01–15.06).

3.15 *LANL data validation reason codes* — The codes applied to the sample data by data validators who are independent of the contract laboratory which performed the sample analysis. Reason codes provide an in-depth and analysis-specific explanation for applying the qualifier with some description of the potential impact on the data use. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate ER Project SOP (ER-SOPs 15.01–15.06).

3.16 *Matrix spike* — An aliquot of sample spiked with a known concentration of target analyte(s). Matrix spike samples are used to measure the ability to recover prescribed analytes from a native sample matrix. The spiking typically occurs before sample preparation and analysis.

3.17 *Nondetect* — Sample result that is less than the MDL. The laboratory reports nondetects as undetected at the EDL.

3.18 *Percent recovery (%R)* — Amount of material detected in a sample (minus any amount already in the sample) divided by the amount added to the sample and expressed as a percentage.

3.19 *Preparation blank* — An analyte-free matrix to which all reagents are added in the same volumes or proportions as those used in the environmental sample processing, and which is prepared and analyzed in the same manner as the corresponding environmental samples. The preparation blank is used to assess the potential for contamination of samples during preparation and analysis.

3.20 *Relative percent difference (RPD)* — The measure used to assess the precision between parent sample results and their associated duplicate results. The RPD is calculated as follows:

$$\left|RPD\right| = \left[\frac{S - R}{\left(\frac{S + R}{2}\right)}\right] 100$$

where RPD = relative percent difference,
S = parent sample result, and
R = duplicate sample result.

The Los Alamos National Laboratory ER Project criteria for the RPD is less than 20% for aqueous samples and less than 35% for soil samples when the sample concentrations are greater than or equal to five times the MDL. For samples with concentrations less than five times the MDL, but greater than the MDL, the control is +/-MDL. No precision criterion applies to samples with concentrations less than the MDL.

3.21 *Request number (RN)* — An identifying number assigned by the ER Project to a group of samples that are submitted for analysis.

3.22 *Routine data* — Data generated using analytical methods that are identified as routine methods in the current ER Project SOW for analytical services.

3.23 *Routine data validation* — Process of reviewing analytical data relative to quantitative routine acceptance criteria. The objectives of routine data validation are to (1) estimate the data's technical quality relative to minimum national guidelines adopted by the ER Project, and (2) indicate to data users

the technical data quality at a gross level by assigning qualifier flags to environmental data whose quality indicators do not meet acceptance criteria.

3.24 *Target analyte* — An element, chemical, or parameter, the concentration, mass, or magnitude of which is designed to be quantified by use of a particular test method.

4.0 BACKGROUND AND PRECAUTIONS

4.1 To protect the integrity of the data record package, the **data validator** must store and handle all data record packages under ER Project chain-of-custody (COC) rules prescribed in ER-SOP-15.09.

4.2 Logic diagrams are included in this SOP to expedite the validation process. Logic diagrams in this SOP do not include instructions about where to record validation results. Those instructions are incorporated in the text that corresponds to each logic diagram.

4.3 The inorganic data validation checklist forms identify actions that must be taken, depending on whether a validation condition is true or false (Attachment D). Look at the top of each data validation checklist to learn the required action.

4.4 This validation process requires that the **validator** record qualifier flags and reason codes on photocopies of the data summary results forms (Form I) in the hard copy data record packages. Contiguous lines of identical qualification on the photocopied Form I may be represented as the qualifier flag and reason code, followed by a vertical downward arrow to the end of the block of results that are qualified identically.

4.5 The inorganic data validation checklist forms in Attachment D are examples of the forms the validator must use to validate data under this SOP. The forms may be reproduced in whole or in part, as needed to complete the validation of a data record package.

5.0 EQUIPMENT

The **validator** may need the following equipment and supplies to implement this procedure:

5.1 current inorganic data validation checklists (see Attachment D),

5.2 data record packages to be validated,

5.3 electronic calculator (optional),

5.4 photocopier, and

5.5 current ER Project SOW for analytical services.

6.0 PROCEDURE

Note: Deviations from SOPs are made in accordance with QP-4.2.

6.1 Prepare for Data Validation

6.1.1 The **validator** will begin by obtaining the required current versions of the inorganic data validation checklist forms (see Attachment D) from the ER Project website (http://erinternal.lanl.gov/Quality/forms.htm).

6.1.2 Obtain from the Sample Management Office (SMO) of the Field Support Facility the data record package(s) that contain the sample data to be validated.

6.1.3 Prepare a data validation cover sheet (see Attachment C) by completing the top part of the form and placing a check or other mark adjacent to the analytical suites for which this validation is being performed.

Note: You may use a single cover sheet when validating multiple analytical suites under the same RN.

Note: Use a separate sheet of paper to document each deficiency identified beyond the scope of this procedure, including phone conversations with the analytical laboratory personnel concerning these deficiencies. Attach these sheets to the data validation cover sheet.

6.1.4 Verify that the following items are present in the data record package:

6.1.4.1 a signed LANL COC record;

6.1.4.2 the case narrative;

6.1.4.3 the result forms (CLP Form I or equivalent) for each sample;

6.1.4.4 the QC forms (CLP forms 2A, 2B, 3, 4, 5A, 5B, 6, 7, 8 [GFAA only], 9 [ICPES only], 10, 11A, 11B, or equivalent) for water and/or soils, as appropriate; and

6.1.4.5 the instrument readout (raw data) for the samples.

6.1.5 If the data record package does not contain all items listed in Sections 6.1.4.1 through 6.1.4.5, contact the analytical laboratory to obtain those materials.

6.1.5.1 If required documentation is missing from the data record package, and the package is less than six months old, contact the analytical laboratory and allow three business days for the laboratory to submit the required documentation.

6.1.5.2 If the analytical laboratory does not submit documentation within three business days, return the data record package to the FSF for contract-compliance action.

6.1.5.3 If the data record package is greater than 6 months old, allow 10 business days for the analytical laboratory to submit the required documentation before returning the data record package to the FSF.

6.1.6 Record the presence or absence (Y or N) of each item, as appropriate, in the completeness checklist of the validation cover sheet.

6.1.7 In the data validation cover sheet completeness checklist section, note any samples whose data are missing from the data record package.

6.1.8 Photocopy all analytical laboratory QC forms from the data record package.

6.1.9 Photocopy the case narrative from the data record package.

6.1.10 Photocopy the form (Form I) that you will use during the validation process before completing the form.

Caution: Do not record data-validation qualifiers and reason codes on the original form (Form I).

Note: The **validator** must submit photocopies of the items listed in Sections 6.1.8 through 6.1.10 as attachments to the completed data validation checklists.

6.2 Detection Status

Note: In order to meet the regulatory requirements imposed upon the ER Project with the technology routinely available from the environmental laboratory community, the ER Project requires analytical laboratories to report inorganic analytes as detected down to the IDL. In order to identify that these results are below the EDL and may have greater errors in quantitation, the laboratories have been instructed to apply a “B” flag to all results between the IDL and EDL. The steps in this section are required to identify results between the IDL and EDL as estimated detections so that the detect status of each analytical result is clear for the validation steps that follow Section 6.2.

6.2.1 If *no* results *are reported* with a “B” flag,

6.2.1.1 record “N” in block 1a of the inorganic data validation checklist, Part I and

6.2.1.2 go to Section 6.3, Verify Calibrations.

6.2.2 For *any* results that *are reported* with a “B” flag,

6.2.2.1 record “Y” in block 1a of the inorganic data validation checklist, Part I;

6.2.2.2 circle “J, I1” in block 1b of the inorganic data validation checklist, Part I; and

6.2.2.3 record the qualifier flag and reason code combination “J, I1” next to each analyte flagged with the “B” flag, on Form I.

6.3 Verify Calibrations

Verify the presence of the initial- and continuing-calibration verification (ICV and CCV) results using the forms supplied by the analytical laboratory.

6.3.1 If an ICV *was analyzed* for each sample matrix and/or analytical batch,

6.3.1.1 record “Y” in block 1a of the inorganic data validation checklist, Part II;

6.3.1.2 record “n/a” in block 1c of the inorganic data validation checklist Part II; and

6.3.1.3 go to Section 6.3.3.

6.3.2 If an ICV *was not analyzed* for each sample matrix and/or analytical batch,

6.3.2.1 record “N” in block 1a of the inorganic data validation checklist, Part II;

6.3.2.2 circle “A, I15a” in block 1b of the inorganic data validation checklist, Part II;

6.3.2.3 record the qualifier flag and reason code combination “A, I15a” next to the affected sample results, on Form I; and

6.3.2.4 record any missing ICV and associated analytes in block 1c of the inorganic data validation checklist, Part II.

6.3.3 If a CCV *was analyzed* for each sample matrix and/or analytical batch,

6.3.3.1 record “Y” in block 2a of the inorganic data validation checklist, Part II;

6.3.3.2 record “n/a” in block 2c of the inorganic data validation checklist, Part II; and

6.3.3.3 go to Section 6.4, Verify Blank Results.

6.3.4 If a CCV *was not analyzed* for each sample matrix and/or analytical batch,

6.3.4.1 record “N” in block 2a of the inorganic data validation checklist, Part II;

6.3.4.2 circle “A, I15b” in block 2b of the inorganic data validation checklist, Part II;

6.3.4.3 record the qualifier flag and reason code combination “A, I15b” next to the affected sample results on the Form I; and

6.3.4.4 record any missing CCV and associated analytes in block 2c of the inorganic data validation checklist, Part II.

6.4 Verify Blank Results

Verify the presence of the initial- and continuing-calibration blanks’ (ICB and CCB) and preparation blanks’ results using forms provided by the analytical laboratory.

Note: The blank results must be compared to the contractually required EDLs. Due to time constraints, only the preparation blank is validated manually.

Note: If additional validation forms are needed to record validation data for more than one blank, make additional copies of the appropriate forms.

6.4.1 If a preparation blank *was analyzed* for each sample matrix and/or analytical batch,

6.4.1.1 record “Y” in block 1a of the inorganic data validation checklist, Part IIIa;

6.4.1.2 record “n/a” in block 1c of the inorganic data validation checklist, Part IIIa; and

6.4.1.3 go to Section 6.4.3.

6.4.2 If a preparation blank *was not analyzed* for each sample matrix and/or analytical batch,

6.4.2.1 record “N” in block 1a of the inorganic data validation checklist, Part IIIa;

6.4.2.2 circle “A, I4b” in block 1b of the inorganic data validation checklist, Part IIIa;

6.4.2.3 record the qualifier flag and reason code combination “A, I4b” next to the results of all samples for which a preparation blank was not analyzed; and

6.4.2.4 record any missing matrices or analytical batches in block 1c of the inorganic data validation checklist, Part IIIa.

6.4.3 If *no* target analytes *were detected* in the preparation blank,

6.4.3.1 record “N” in blocks 2a and 3a of the inorganic data validation checklist, Part IIIb;

6.4.3.2 record “n/a” in blocks 2c, 3c, 2d, and 3d of the inorganic data validation checklist, Part IIIb; and

6.4.3.3 go to Section 6.5, Verify ICPES Interference Check Sample Results.

6.4.4 If target analytes *were detected* in the preparation blank *and* none of the target analytes detected in the preparation blank were detected in any sample,

6.4.4.1 record “N” in blocks 2a and 3a of the inorganic data validation checklist, Part IIIb;

6.4.4.2 record “n/a” in blocks 2c, 3c, 2d, and 3d of the inorganic data validation checklist, Part IIIb; and

6.4.4.3 go to Section 6.5, Verify ICPES Interference Check Sample Results.

6.4.5 If any target analytes *were detected* in the preparation blank *and* the *same* target analytes were detected in the sample at a level greater than five times the amount detected in the preparation blank,

6.4.5.1 record “N” in blocks 2a and 3a of the inorganic data validation checklist, Part IIIb;

6.4.5.2 record “n/a” in blocks 2c, 3c, 2d, and 3d of the inorganic data validation checklist, Part IIIb; and

6.4.5.3 go to Section 6.5, Verify ICPES Interference Check Sample Results.

6.4.6 If target any analytes *were detected* in the preparation blank *and* the *same* target analytes were detected in the sample at a level less than the EDL *and* were less than or equal to five times the amount detected in the preparation blank,

6.4.6.1 record “Y” in block 2a of the inorganic data validation checklist, Part IIIb;

6.4.6.2 circle “U, I5” in block 2b of the inorganic data validation checklist, Part IIIb;

6.4.6.3 record the qualifier flag and reason code combination "U, I5" next to the result for each affected target analyte, on Form I;

6.4.6.4 record which samples and analytes have been qualified "U" in block 2c of the inorganic data validation checklist, Part IIIb; and

6.4.6.5 record the analyte names and their blank concentrations, for all analytes detected in the blank, in block 2d of the inorganic data validation checklist, Part IIIb.

6.4.7 If any target analytes *were detected* in the preparation blank *and* the *same* target analytes were detected in the sample at a level greater than the EDL *and* were less than or equal to five times the amount detected in the preparation blank,

6.4.7.1 record "Y" in block 3a of the inorganic data validation checklist, Part IIIb;

6.4.7.2 circle "U, I4a" in block 3c of the inorganic data validation checklist, Part IIIb;

6.4.7.3 record the qualifier flag and reason code combination "U, I4a" next to the result for each affected target analyte, on Form I;

6.4.7.4 record which samples and analytes have been qualified "U" in block 3c of the inorganic data validation checklist, Part IIIb; and

6.4.7.5 record the analyte names and their blank concentrations, for all analytes detected in the blank, in block 3d of the inorganic data validation checklist, Part IIIb.

6.4.8 Use the logic diagram in Figure 6.4-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant preparation blanks.

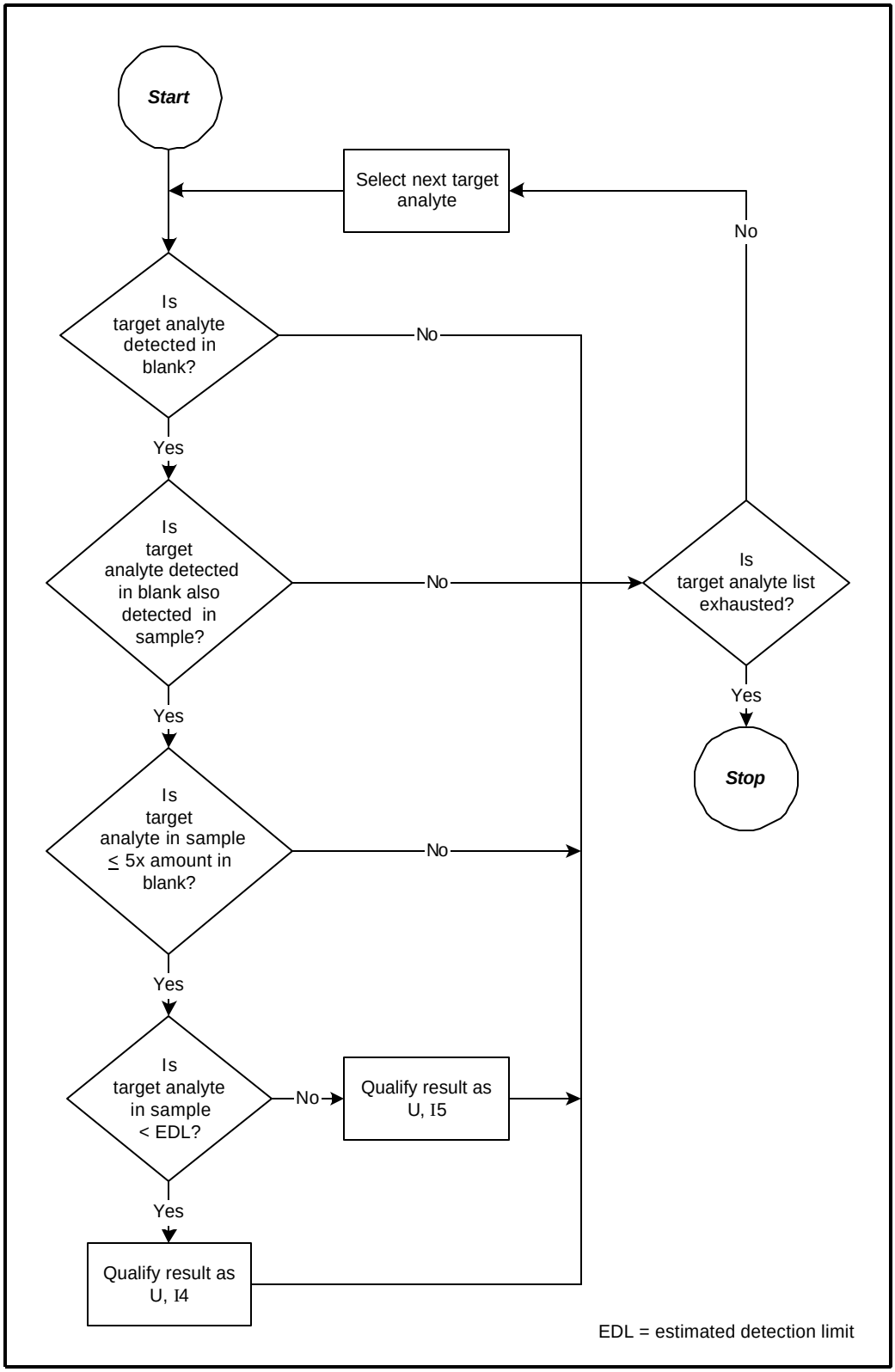

Figure 6.4-1. Assigning LANL qualifier flags and reason codes to the sample results for noncompliant preparation blanks.

6.5 Verify ICPES Interference Check Sample Results

Verify the presence of the ICS %R values using forms provided by the analytical laboratory. The ICS must contain the following analytes: Ag, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb (see note), V and Zn. The QC acceptance limits are ± 20%.

Note: If lead was analyzed by graphite furnace atomic absorption (GFAA), no ICS result is required. This information may be noted in the inorganic data validation checklist Part IVa, block 1c.

6.5.1 If an ICS *was analyzed* for each sample matrix and/or analytical batch,

6.5.1.1 record “Y” in block 1a of the inorganic data validation checklist, Part IVa;

6.5.1.2 record “n/a” in block 1c of the inorganic data validation checklist, Part IVa; and

6.5.1.3 go to Section 6.5.3.

6.5.2 If an ICS *was not analyzed* for each sample matrix and/or analytical batch,

6.5.2.1 record “N” in block 1a of the inorganic data validation checklist, Part IVa;

6.5.2.2 circle “A, I7d” in block 1b of the inorganic data validation checklist, Part IVa;

6.5.2.3 record the qualifier flag and reason code combination “A, I7d” next to all associated sample analytes for which the ICS was not analyzed, on the Form I; and

6.5.2.4 record any missing ICS analytes in block 1c of the inorganic data validation checklist, Part IVa.

6.5.3 If *all* ICS analyte %R values are greater than or equal to 80% *and* less than or equal to 120%,

6.5.3.1 record “N” in blocks 2a, 3a, and 4a of the inorganic data validation checklist, Part IVb;

6.5.3.2 record “n/a” in blocks 2c, 3c, and 4c of the inorganic data validation checklist, Part IVb; and

6.5.3.3 go to Section 6.6, Verify Matrix-Spike Results.

6.5.4 If *any* ICS analyte %R value is greater than 120%,

6.5.4.1 record “Y” in block 2a of the inorganic data validation checklist, Part IVb.

6.5.4.2 For each *detected* analyte in the sample,

1) circle “J+, I7” in block 2b of the inorganic data validation checklist, Part IVb;

2) record the qualifier flag and reason code combination “J+, I7” next to the result for each affected target analyte, on Form I; and

3) record the affected samples and analytes in block 2c of the inorganic data validation checklist, Part IVb.

6.5.4.3 For each *nondetected* analyte in the sample,

1) record “n/a” in block 2c of the inorganic data validation checklist, Part IVb.

6.5.5 If *any* ICS analyte %R value is greater than or equal to 50% *and* less than 80%,

6.5.5.1 record “Y” in block 3a of the inorganic data validation checklist, Part IVb.

6.5.5.2 For each *detected* analyte in the sample,

1) circle “J-, I7a” in block 3b of the inorganic data validation checklist, Part IVb;

2) record the qualifier flag and reason code combination “J-, I7a” next to the result for each affected target analyte, on Form I; and

3) record the affected samples and analytes in block 3c of the inorganic data validation checklist, Part IVb.

6.5.5.3 For each *nondetected* analyte in the sample,

1) circle “UJ, I7c” in block 3b of the inorganic data validation checklist, Part IVb;

2) record the qualifier flag and reason code combination “UJ, I7c” next to the result for each affected target analyte, on Form I; and

3) record the affected samples and analytes in block 3c of the inorganic data validation checklist, Part IVb.

6.5.6 If *all* ICS %R values are greater than 50%,

6.5.6.1 record “N” in block 4a of the inorganic data validation checklist, Part IVb;

6.5.6.2 record “n/a” in block 4c of the inorganic data validation checklist, Part IVb; and

6.5.6.3 go to Section 6.6, Verify Matrix-Spike Results.

6.5.7 If *any* ICS analyte %R value is less than 50%,

6.5.7.1 record “Y” in block 4a of the inorganic data validation checklist, Part IVb.

6.5.7.2 For *all* analytes that correspond to the noncompliant ICS,

1) circle “RPM, I7b” in block 4b of the inorganic data validation checklist, Part IVb;

2) record the qualifier flag and reason code combination “RPM, I7b” next to the result for each affected target analyte, on Form I; and

3) record the affected samples and analytes in block 4c of the inorganic data validation checklist Part IVb.

6.5.8 Use the logic diagram in Figure 6.5-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant ICS analytes.

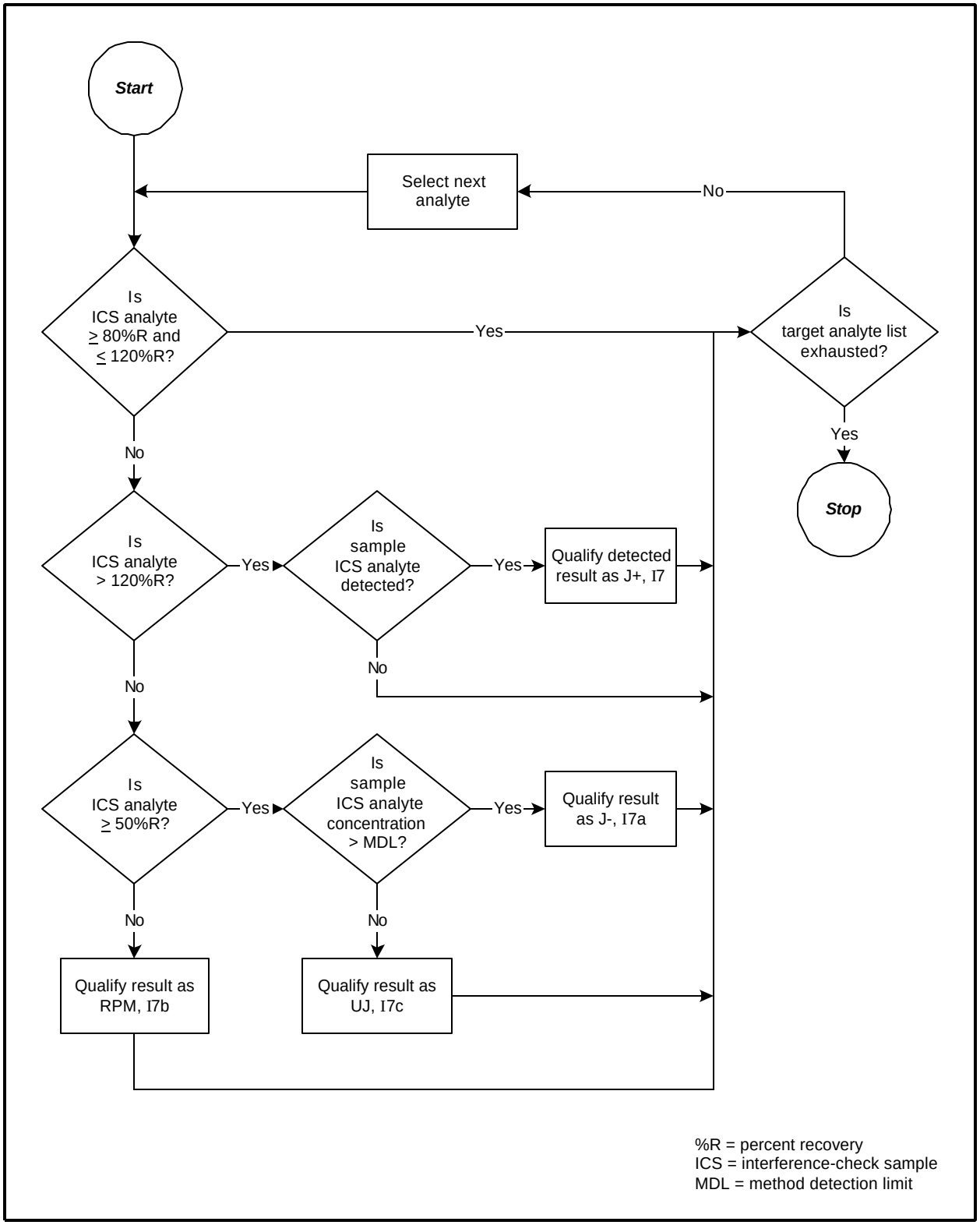

Figure 6.5-1. Assigning LANL qualifier flags and reason codes to the sample results for noncompliant ICS analytes.

6.6 Verify Matrix-Spike Results

Verify the presence of the matrix spike sample %R values using the forms provided by the analytical laboratory. The matrix-spike acceptance criteria are 75%–125%, inclusive, for all spiked analytes.

Note: See the matrix-spike requirements table in the inorganic data validation checklist Part Vc. If the sample result is greater than four times the spike added, these acceptance criteria do not apply.

6.6.1 If a matrix spike *was analyzed* on a sample associated with this request *and* the matrix spike included all required analytes,

6.6.1.1 record "Y" in block 1a of the inorganic data validation checklist, Part Va;

6.6.1.2 record "n/a" in block 1c of the inorganic data validation checklist, Part Va; and

6.6.1.3 go to Section 6.6.4.

6.6.2 If a matrix spike *was analyzed* on a sample *not associated* with this request *and* no matrix spike was analyzed on a sample associated with this request,

6.6.2.1 record "N" in block 1a of the inorganic data validation checklist, Part Va;

6.6.2.2 circle "PM, I14b" in block 1b of the inorganic data validation checklist, Part Va;

6.6.2.3 record the qualifier flag and reason code combination "PM, I14b" next to the result for each affected target analyte, on Form I;

6.6.2.4 if the matrix-spike sample *was* a ER Project sample, record the RN and sample ID of the spike sample in block 1c of the inorganic data validation checklist, Part Va; and

6.6.2.5 if the matrix-spike sample *was not* a ER Project sample, note this in block 1c of the inorganic data validation checklist, Part Va.

6.6.3 If insufficient sample volume was submitted for analysis and *no* matrix spike could be analyzed,

6.6.3.1 record "N" in block 1a of the inorganic data validation checklist, Part Va;

6.6.3.2 circle "A, I14a" in block 1b of the inorganic data validation checklist, Part Va; and

6.6.3.3 record the qualifier flag and reason code combination "A, I14a" next to the result for each affected target analyte, on Form I.

6.6.4 If *all* matrix-spike %R values meet the acceptance criteria (75%–125%) inclusive,

6.6.4.1 record "N" in blocks 2a, 3a, 4a, and 5a of the inorganic data validation checklist, Part Vb;

6.6.4.2 record "n/a" in blocks 2c, 2d, 3c, 3d, 4c, 4d, 5c and 5d of the inorganic data validation checklist, Part Vb; and

6.6.4.3 go to Section 6.7, Verify Duplicate-Sample Results.

6.6.5 If *all* matrix-spike %R values are less than or equal to 150%,

6.6.5.1 record "N" in block 2a of the inorganic data validation checklist, Part Vb;

6.6.5.2 record "n/a" in blocks 2c and 2d of the inorganic data validation checklist, Part Vb; and

6.6.5.3 go to Section 6.6.7.

6.6.6 If *any* matrix-spike %R value is greater than 150%,

6.6.6.1 record "Y" in block 2a of the inorganic data validation checklist, Part Vb.

6.6.6.2 For each *detected* sample result,

1) circle "J+, I3h" in block 2b of the inorganic data validation checklist, Part Vb;

2) record the qualifier flag and reason code combination "J+, I3h" next to all affected analytes, on Form I;

3) record the analytes that do not meet the criteria and the affected samples in block 2c of the inorganic data validation checklist, Part Vb; and

4) record the %R values of the analytes that do not meet the acceptance criterion in block 2d of the inorganic data validation checklist, Part Vb.

6.6.6.3 For each *nondetected* sample result,

1) circle "UJ, I3g" in block 2b of the inorganic data validation checklist, Part Vb;

2) record the qualifier flag and reason code combination "UJ, I3g" for affected analytes, on Form I;

3) record the analytes that do not meet the criteria and the affected samples in block 2c of the inorganic data validation checklist, Part Vb;

4) record the %R values of the analytes that do not meet the acceptance criterion in block 2d of the inorganic data validation checklist, Part Vb.

6.6.7 If *all* matrix-spike %R values are less than or equal to 125%,

6.6.7.1 record "N" in block 3a of the inorganic data validation checklist, Part Vb;

6.6.7.2 record "n/a" in blocks 3c and 3d of the inorganic data validation checklist, Part Vb; and

6.6.7.3 go to Section 6.6.9.

6.6.8 If *any* matrix-spike %R value is greater than 125% and less than or equal to150%,

6.6.8.1 and the target analyte *is detected* in the sample,

1) record "Y" in block 3a of the inorganic data validation checklist, Part Vb;

2) circle "J+, I3" in block 3b of the inorganic data validation checklist, Part Vb;

3) record the qualifier flag and reason code combination "J+, I3" next to the result for each affected target analyte, on Form I;

4) record the analytes that do not meet the acceptance criterion and the affected samples in block 3c of the inorganic data validation checklist, Part Vb; and

5) record the %R values of the analytes that do not meet the acceptance criterion in block 3d of the inorganic data validation checklist, Part Vb;

6.6.8.2 and the target analyte *is not detected* in the sample,

1) record "Y" in block 3a of the inorganic data validation checklist, Part Vb and

2) record "n/a" in blocks 3c and 3d of the inorganic data validation checklist, Part Vb.

6.6.9 If *no* matrix-spike %R values *are in* the range of greater than or equal to 30% and less than 75%,

6.6.9.1 record “N” in block 4a of the inorganic data validation checklist, Part Vb;

6.6.9.2 record “n/a” in blocks 4c and 4d of the inorganic data validation checklist, Part Vb; and

6.6.9.3 go to Section 6.6.11.

6.6.10 If *any* matrix-spike %R value is greater than or equal to 30% *and* is less than 75%,

6.6.10.1 record “Y” in block 4a of the inorganic data validation checklist Part Vb.

6.6.10.2 For each *detected* analyte in the sample,

1) circle “J-, I3a” in block 4b of the inorganic data validation checklist, Part Vb;

2) record the qualifier flag and reason code combination “J-, I3a” next to the result for each affected target analyte, on Form I;

3) record the analytes that do not meet the acceptance criterion and the affected samples in block 4c of the inorganic data validation checklist, Part Vb; and

4) record the %R values of the analytes that do not meet the acceptance criterion in block 4d of the inorganic data validation checklist, Part Vb.

6.6.10.3 For each *nondetected* analyte in the sample,

1) circle “UJ, I3e” in block 4b of the inorganic data validation checklist, Part Vb;

2) record the qualifier flag and reason code combination “UJ, I3e” next to the result for each affected target analyte, on Form I;

3) record the analytes that do not meet the acceptance criterion and affected samples in block 4c of the inorganic data validation checklist, Part Vb; and

4) record the %R values of the analytes that do not meet the acceptance criterion in block 4d of the inorganic data validation checklist, Part Vb.

6.6.11 If *no* matrix-spike %R value is less than 30%,

6.6.11.1 record “N” in block 5a of the inorganic data validation checklist, Part Vb and

6.6.11.2 record “n/a” in blocks 5c and 5d of the inorganic data validation checklist, Part Vb.

6.6.12 If any matrix-spike %R value is less than 30%,

6.6.12.1 record a “Y” in block 5a of the inorganic data validation checklist Part Vb.

6.6.12.2 For each *detected* analyte in the sample,

1) circle “J-, I3f” in block 5b of the inorganic data validation checklist, Part Vb;

2) record the qualifier flag and reason code combination “J-, I3f” next to each affected analyte, on Form I;

3) record the analytes that do not meet the acceptance criterion and the affected samples in block 5c of the inorganic data validation checklist, Part Vb; and

4) record the %R values of the analytes that do not meet the acceptance criterion in block 5d of the inorganic data validation checklist, Part Vb.

6.6.12.3 For each *nondetected* analyte in the sample,

1) circle “RPM, I3d” in block 5b of the inorganic data validation checklist, Part Vb;

2) record the qualifier flag and reason code combination “RPM, I3d” next to each affected analyte, on Form I;

3) record the analytes that do not meet the acceptance criterion and the affected samples in block 5c of the inorganic data validation checklist, Part Vb; and

4) record the %R values of the analytes that do not meet the acceptance criterion in block 5d of the inorganic data validation checklist, Part Vb.

6.6.13 Use the logic diagram in Figure 6.6-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant matrix-spike analytes.

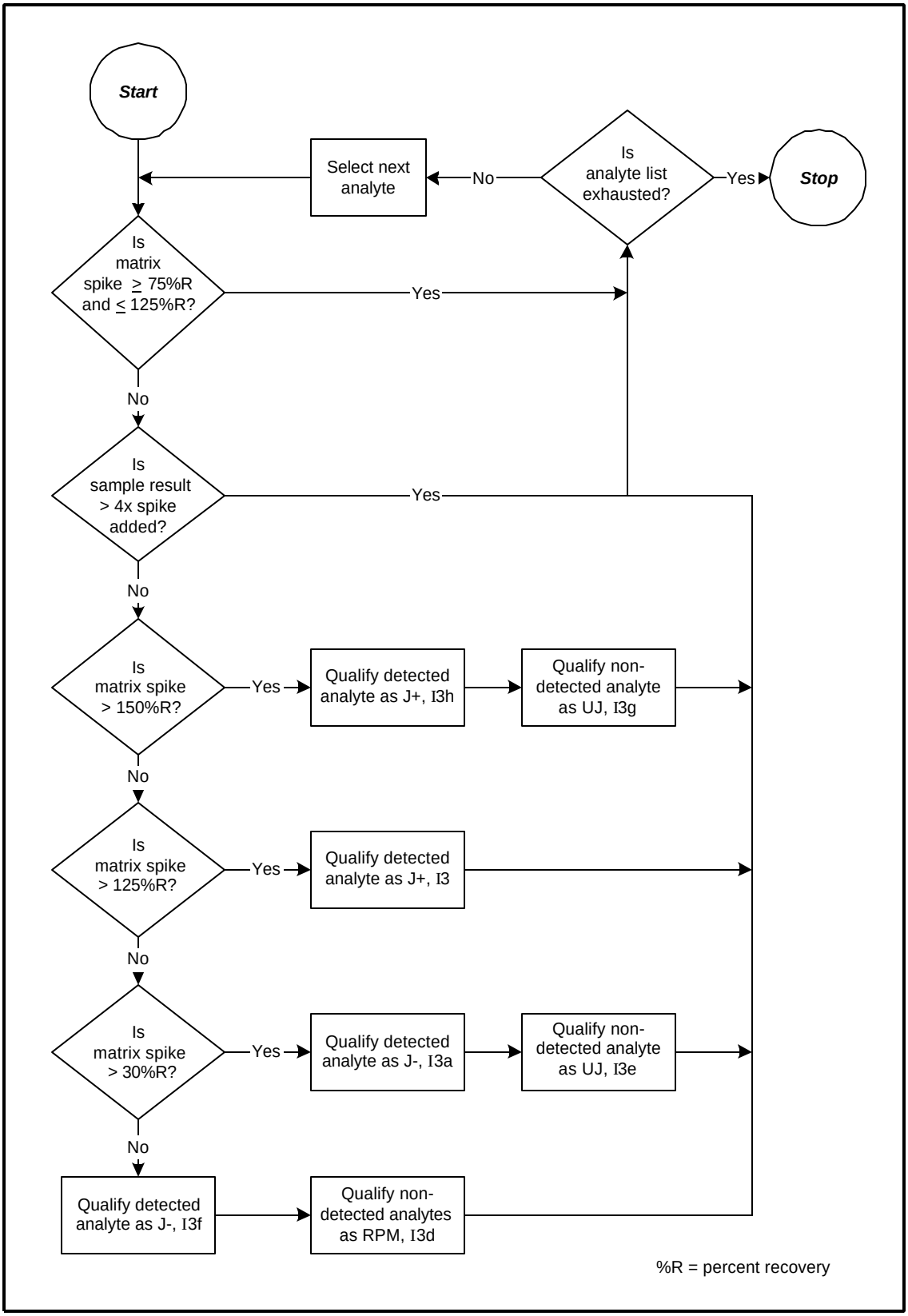

Figure 6.6-1. Assigning LANL qualifier flags and reason codes to the sample results for noncompliant matrix-spike analytes.

6.7 Verify Duplicate-Sample Analysis Results

Verify the presence of the analytical laboratory duplicate-sample %R values using the forms provided by the analytical laboratory. If the sample and duplicate-sample results are greater than or equal to five times the EDL, the duplicate-sample criterion for aqueous samples is a RPD less than or equal to 20%; the duplicate-sample criterion for solid samples is a RPD less than or equal to 35%. If either the sample or duplicate-sample value is less than five times the EDL, a control limit must be used: equal to the EDL for water samples and two times the EDL for solid samples.

6.7.1 If a duplicate sample *was analyzed* on a sample *associated* with this request and the duplicate-sample analysis included all required analytes,

6.7.1.1 record “Y” in block 1a of the inorganic data validation checklist, Part VIa;

6.7.1.2 record “n/a” in block 1c of the inorganic data validation checklist, Part VIa; and

6.7.1.3 go to Section 6.7.4.

6.7.2 If a duplicate sample *was analyzed* on a sample *not associated* with this request,

6.7.2.1 record “N” in block 1a of the inorganic data validation checklist, Part VIa;

6.7.2.2 circle “PM, I13b” in block 1b of the inorganic data validation checklist, Part VIa;

6.7.2.3 record the qualifier flag and reason code combination “PM, I13b” next to the result for each affected target analyte, on Form I;

6.7.2.4 if the duplicated sample *was* an ER Project sample associated with another ER Project RN, record the RN and sample ID of the duplicated sample in block 1c of the inorganic data validation checklist, Part VIa;

6.7.2.5 if the duplicated sample *was not* an ER Project sample, note this in block 1c of the inorganic data validation checklist, Part VIa; and

6.7.2.6 Go to Section 6.7.4.

6.7.3 If insufficient sample volume was submitted for analysis and *no* duplicate sample could be analyzed,

6.7.3.1 record “N” in block 1a of the inorganic data validation checklist, Part VIa;

6.7.3.2 circle “A, I13a” in block 1b of the inorganic data validation checklist, Part VIa;

6.7.3.3 record the qualifier flag and reason code combination “A, I13a” next to the result for each affected target analyte, on the Form I;

6.7.3.4 record any analytes or samples that are associated with the missing duplicate sample in block 1c of the inorganic data validation checklist, Part VIa;

6.7.3.5 record “n/a” in blocks 2a, 2c, 2d, 3a, 3c, and 3d of the inorganic data validation checklist, Parts VIb and VIc; and

6.7.3.6 go to Section 6.8, Verify Laboratory Control Sample Results.

6.7.4 If *neither* the sample result *nor* the duplicate-sample result is less than five times the EDL,

6.7.4.1 and if *all* duplicate-sample RPDs *meet* the criteria (listed in Section 6.7),

1) record “Y” in block 2a of the inorganic data validation checklist, Part VIb;

2) record “n/a” in blocks 2c and 2d of the inorganic data validation checklist, Part VIb.

6.7.5 If *neither* the sample result *nor* the duplicate-sample result is less than five times the EDL,

6.7.5.1 For *each* duplicate-sample RPD that *exceeds* 20% for aqueous samples or 35% for soil samples,

1) record “N” in block 2a of the inorganic data validation checklist, Part VIb;

2) circle, in block 2b of the inorganic data validation checklist, Part VIb,

- “J, I10” for *detected* analytes and
- “UJ, I10a” for *nondetected* analytes;

3) record, next to the result for each affected target analyte, on Form I, the qualifier flag and reason code combination

- "J, I10" for *detected* analytes and
- "UJ, I10a" for *nondetected* analytes;

4) record any analytes that do not meet the acceptance criteria and the affected samples in block 2c of the inorganic data validation checklist, Part VIb; and

5) record the RPDs of the analytes that do not meet the acceptance criteria and the affected samples in block 2d of the inorganic data validation checklist, Part VIb.

6.7.6 If *either* the sample result *or* the duplicate-sample result are less than five times the EDL, record

6.7.6.1 "Y" in block 2a of the inorganic data validation checklist, Part VIb and

6.7.6.2 "n/a" in blocks 2c and 2d of the inorganic data validation checklist, Part VIb.

6.7.6.3 And if *all* duplicate-sample RPDs *meet* the criteria, record

1) "Y" in block 3a of the inorganic data validation checklist, Part VIc and

2) "n/a" in blocks 3c and 3d of the inorganic data validation checklist, Part VIc.

6.7.6.4 Or if the difference between the sample result and the duplicate-sample result is greater than the EDL for water samples or greater than two times the EDL for solid samples,

1) record "N" in block 3a of the inorganic data validation checklist, Part VIc;

2) circle, in block 3b of the inorganic data validation checklist, Part VIc,

- "J, I10" for *detected* analytes and
- "UJ, I10a" for *nondetected* analytes;

3) record, next to the result for each affected target analyte, on Form I, the qualifier flag and reason code combination

- "J, I10" for *detected* analytes and
- "UJ, I10a" for *nondetected* analytes;

4) record the analytes that do not meet the acceptance criteria and the affected samples in block 3c of the inorganic data validation checklist, Part VIc; and

5) record the RPDs of the analytes that do not meet the acceptance criteria and the affected samples in block 3d of the inorganic data validation checklist, Part VIc.

6.7.7 Use the logic diagram in Figure 6.7-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant duplicate-sample analytes.

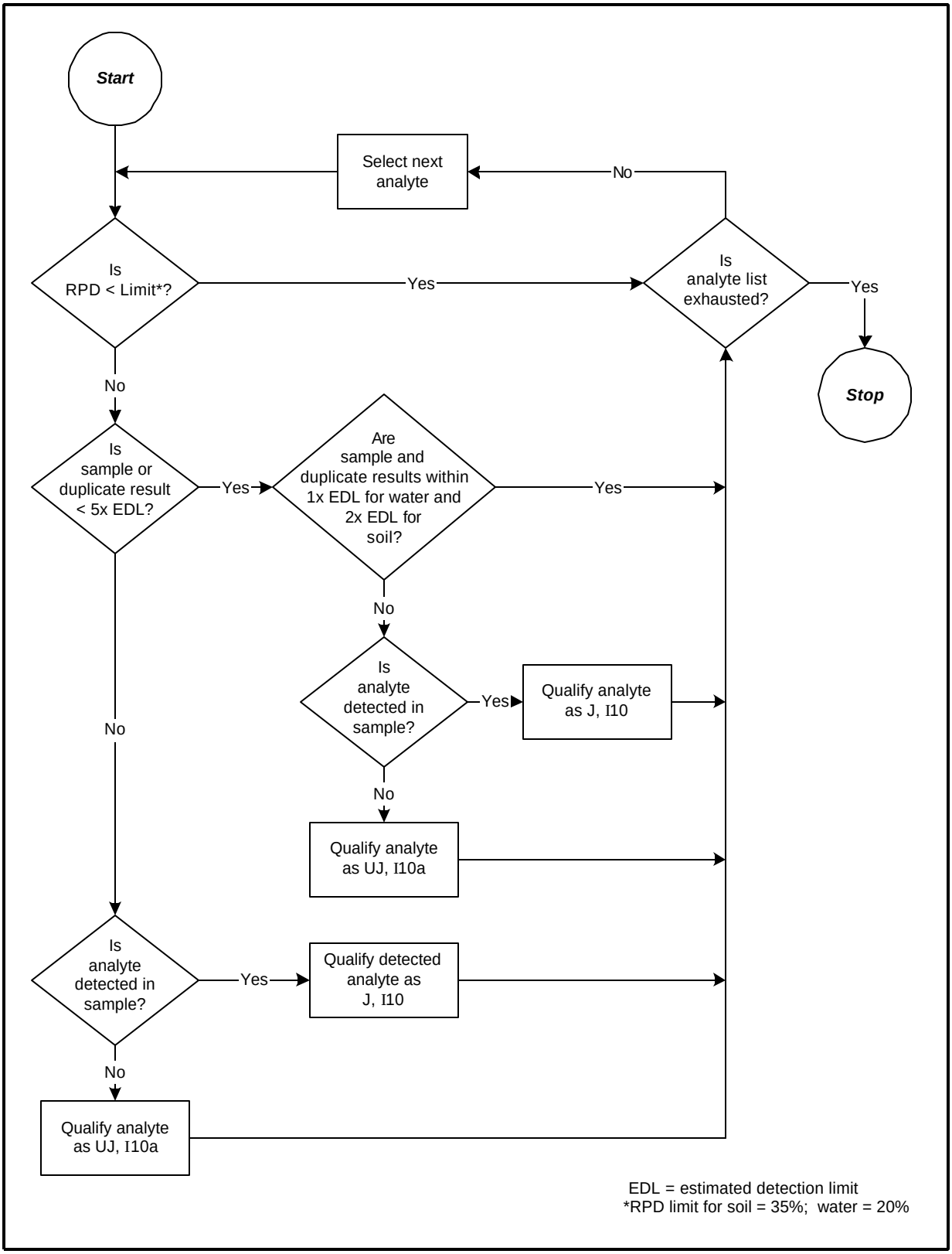

Figure 6.7-1. Assigning LANL qualifier flags and reason codes to the sample results for noncompliant duplicate-sample analytes.

6.8 Verify Laboratory Control Sample Results

Verify the presence of the LCS sample %R values using forms provided by the analytical laboratory. An aqueous LCS must be prepared and analyzed for each aqueous RN. For soil samples, if a solid LCS is available from a vendor, then a solid LCS should be used for those samples. However, there are no acceptance criteria for solid LCS samples. If a solid LCS is unavailable, an aqueous LCS must be used and the aqueous LCS criteria are used to evaluate the results. The following sections describe how sample results are qualified based on the aqueous LCS results.

Note: The solid LCS results may be compared to the aqueous criteria and then any analytes that do not meet the criteria should be noted in block 1c of the inorganic data validation checklist Part VII. No qualification is applied to solid LCS results that do not meet aqueous criteria as this information is provided only to aid in assessing possible data quality.

6.8.1 If an appropriate LCS *was analyzed* for *all* samples in this RN,

6.8.1.1 record “N” in block 1a, of the inorganic data validation checklist, Part VII;

6.8.1.2 record “n/a” in block 1c of the inorganic data validation checklist Part VII; and

6.8.1.3 go to Section 6.8.3.

6.8.2 If an appropriate LCS *was not analyzed* for *any* sample in this RN,

6.8.2.1 record “Y” in block 1a, of the inorganic data validation checklist, Part VII;

6.8.2.2 circle “A, I6c” in block 1b of the inorganic data validation checklist, Part VII;

6.8.2.3 record the qualifier flag and reason code combination “A, I6c” next to the result for each affected target analyte in each affected sample, on Form I; and

6.8.2.4 record the LCS or LCS analytes that were not analyzed with this RN in block 1c of the inorganic data validation checklist, Part VII.

6.8.3 If the LCS %R values for *all* analytes (except for Ag and Sb) fall between 80%–120%, inclusive,

6.8.3.1 record “N” in blocks 2a and 3a of the inorganic data validation checklist, Part VII;

6.8.3.2 record “n/a” in blocks 2c, 2d, 3c, and 3d of the inorganic data validation checklist, Part VII; and

6.8.3.3 go to Section 6.9, Verify Holding Times.

6.8.4 If *no* LCS analyte %R value is less than 80%,

6.8.4.1 record "N" in block 2a of the inorganic data validation checklist, Part VII;

6.8.4.2 record "n/a" in blocks 2c and 2d of the inorganic data validation checklist, Part VII; and

6.8.4.3 go to Section 6.8.6.

6.8.5 If *any* LCS analyte %R value is less than 80%,

6.8.5.1 record "Y" in block 2a of the inorganic data validation checklist, Part VII;

6.8.5.2 circle, in block 2b of the inorganic data validation checklist, Part VII,

1) "J-, I6a" for *detected* sample analytes and

2) "UJ, I6b" for *nondetected* analytes;

6.8.5.3 record, next to the result for each affected target analyte, on the Form I, the qualifier flag and reason code combination

1) "J-, I6a" for *detected* sample analytes and

2) "UJ, I6b" for *nondetected* analytes;

6.8.5.4 record the noncompliant LCS analytes in block 2c of the inorganic data validation checklist, Part VII; and

6.8.5.5 record the %R values of the noncompliant LCS analytes in block 2d of the inorganic data validation checklist, Part VII.

6.8.6 If *no* LCS analyte %R value is greater than 120%,

6.8.6.1 record "N" in block 3a of the inorganic data validation checklist, Part VII;

6.8.6.2 record "n/a" in blocks 3c and 3d of the inorganic data validation checklist, Part VII; and

6.8.6.3 go to Section 6.9, Verify Holding Times.

6.8.7 If *any* LCS analyte %R value is greater than 120%,

6.8.7.1 record "Y" in block 3a of the inorganic data validation checklist, Part VII;

6.8.7.2 circle "J+, I6" for *detected* sample analytes in block 3b of the inorganic data validation checklist, Part VII;

6.8.7.3 record the qualifier flag and reason code combination "J+, I6" next to the result for *detected* analytes for each affected target analyte, on Form I;

6.8.7.4 record the noncompliant LCS analytes in block 3c of inorganic data validation checklist, Part VII; and

6.8.7.5 record the %R values of the noncompliant LCS analytes in block 3d of inorganic data validation checklist, Part VII.

6.8.8 Use the logic diagram in Figure 6.8-1 to determine which, if any, LANL qualifier flags and reason codes the **validator** must assign to the sample results for noncompliant LCS analytes.

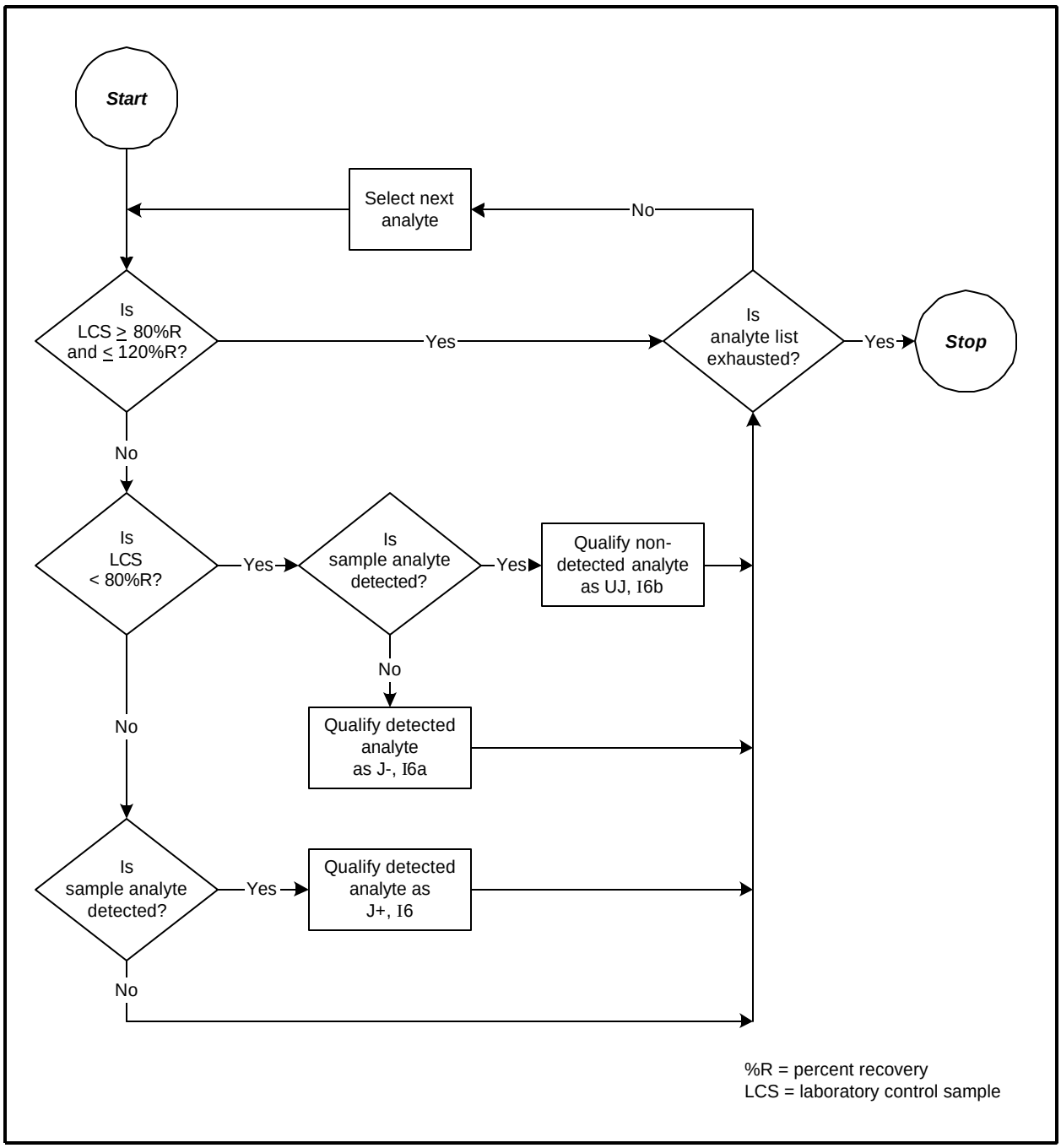

Figure 6.8-1. Assigning LANL qualifier flags and reason codes to the sample results for noncompliant LCS analytes.

6.9 Verify Holding Times

Note: Because holding times for metals (except mercury) are typically six months, holding times are only checked for mercury and cyanide in aqueous samples. The holding time for mercury is 28 days after sample collection. The holding time for cyanide is 14 days after sample collection. Applicable

storage conditions are found in the current SOW for analytical services (LANL 1995).

6.9.1 If *all* samples *were analyzed* within the prescribed holding time,

6.9.1.1 record “Y” in block 1a of the inorganic data validation checklist, Part VIII;

6.9.1.2 record “n/a” in blocks 1c and 1d of the inorganic data validation checklist, Part VIII; and

6.9.1.3 go to Section 6.10, Assemble the Data Record Package.

6.9.2 If any sample *was not analyzed* within the prescribed holding time,

6.9.2.1 record “N” in block 1a of the inorganic data validation checklist, Part VIII;

6.9.2.2 circle “PM, I9” for affected analytes in block 1b of the inorganic data validation checklist, Part VIII;

6.9.2.3 record the qualifier flag and reason code combination “PM, I9” for all affected samples/analytes, on Form I;

6.9.2.4 record the sample ID in block 1c of the inorganic data validation checklist, Part VIII; and

6.9.2.5 record the number of days by which the holding time was exceeded in block 1d of the inorganic data validation checklist, Part VIII.

6.10 Assemble the validation data record package to include the following items in the order they are listed below:

6.10.1 the completed, signed, and dated Data Validation Cover Sheet;

6.10.2 the inorganic data validation checklists completed in Sections 6.2 through 6.9;

6.10.3 photocopies of the completed forms (Form I) on which the validator recorded data validation qualifier flags and reason codes;

6.10.4 a photocopy of the data record package case narrative;

6.10.5 photocopies of the data record package QC forms (assemble in order by QC forms).

6.11 Submit the validation data record package to the FSF, in accordance with ER-SOP-15.09.

7.0 REFERENCES

The following documents have been cited within this procedure.

EPA (US Environmental Protection Agency), February 1994. “US EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review,” Publication 9240.1-05-01, EPA-540/R-94/013, Office of Solid Waste and Emergency Response, Washington, DC.

ER-SOP-15.09, Chain of Custody for Analytical Data Packages

LANL (Los Alamos National Laboratory), July 1995. “Environmental Restoration Project Statement of Work for Analytical Services,” Revision 2, RFP Number 9-SX1-Q4257, Los Alamos National Laboratory, Los Alamos, New Mexico.

QP-2.2, Personnel Orientation and Training

QP-4.2, Standard Operating Procedure Development

8.0 RECORDS

Although no records will be submitted to the Records Processing Facility (RPF) in the course of completing this procedure, the items identified in Section 6.11 will be a part of the data record package submitted to the RPF from the FSF in accordance with ER-SOP-15.09.

9.0 ATTACHMENTS

The document user may employ documentation formats different from those attached to/named in this procedure—as long as the substituted formats in use provide, as a minimum, the information required in the official forms developed by the procedure.

Attachment A: Laboratory Data Validation Qualifier Flags (1 page)

Attachment B: Inorganic Data Validation Reason Codes (2 pages)

Attachment C: Data Validation Cover Sheet (1 page)

Attachment D: Inorganic Data Validation Checklists, Part I through Part VIII (11 Pages)

Laboratory Data Validation Qualifier Flags

A The contractually required supporting documentation for this datum is absent.

U The analyte is classified as “not detected.”

J The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual.

J+ The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual with a potential positive bias.

J- The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual with a potential negative bias.

UJ The analyte is classified as “not detected” with an expectation that the reported result is more uncertain than usual.

RPM The reported sample result is classified as “rejected” due to serious non-compliances regarding quality control acceptance criteria. The presence or absence of the analyte cannot be verified based on routine validation alone.

PM Manual review of raw data is recommended to determine if the observed non-compliance(s) with quality acceptance criteria adversely impacts data use.

Note: A “PM” qualifier flag indicates that a manual review should be conducted if the datum that is qualified with the “PM” is important to the data user. In addition, “PM” also means that a decision must be made by the project manager/delegee regarding the need for further review of the data. This review should include some consideration of potential impact that could result from using the “PM” qualified data.

Inorganic Data Validation Reason Codes

I1 The sample result was reported as detected between the IDL and the EDL. Reported result may be less precise than results which are reported as being above the EDL.

I3 The spike percent recovery value is greater than or equal to the upper acceptance limit (125%) but less than or equal to 150% and the result is a detect, which indicates a potential high bias in the sample results.

I3a The spike percent recovery value is greater than 30% *and* less than the lower acceptance limit (75%), and the sample result is a detect, which indicates a potential low bias in the results.

I3d The spike percent recovery value is less than 30%, and the result is a nondetect, which increases the potential for false negatives being reported. This could be caused by analytical interferences.

I3e The spike percent recovery value is greater than 30% *and* less than the lower acceptance limit (75%), and the sample result is a nondetect, which indicates a potential for false negatives being reported.

I3f The spike percent recovery value is less than 30% and the sample result is a detect, which indicates a potential low bias.

I3g The sample result is undetected and the spike percent recovery value is greater than 150%, which indicates a potential bias in the sample result.

I3h The sample result is detected and the spike percent recovery value is greater than 150%, which indicates a potential high bias in the sample result.

I4a In comparison with the preparation blank, the sample result is greater than the EDL but less than or equal to five times the concentration of the related analyte in the blank.

I4b Preparation blank data was not reported by the analytical laboratory.

I5 The sample result is less than the EDL and is considered to be not detected.

I6 The percent recovery value of the analyte in the LCS is greater than the upper acceptance limit, which indicates a potential for quantitation problems in the analyses and the potential for false positive results being reported.

I6a The percent recovery value of the analyte in the LCS is less than the lower acceptance limit and the analyte is a detect, which indicates a potential for quantitation problems in the analyses and the potential for false negative results being reported.

I6b The percent recovery value of the analyte in the LCS is less than the lower acceptance limit and the analyte is a nondetect, which indicates a potential for

quantitation problems in the analyses and the potential for false negative results being reported.

I6c The corresponding LCS or LCS analyte was not analyzed with the associated batch.

I7 The ICS percent recovery value is greater than 120% and the result is a detect, which indicates potential quantitation problems in the analyses and the potential for false positive results being reported.

I7a The ICS percent recovery value is greater than or equal to 50% *and* less than 80% and the result is a detect, which indicates a potential for a low bias.

I7b The ICS percent recovery value is less than 50%, which indicates a greatly increased potential for false negative sample results being reported.

I7c The ICS percent recovery value is greater than or equal to 50% and less than 80%, and result is a nondetect, which indicates a potential for false negative results being reported.

I7d The ICS data was not provided by the analytical laboratory.

I9 The holding time is exceeded. Positive results may be biased low and nondetected analytes may be false negatives. An evaluation of the data with respect to the technical implications of exceeding the holding time is recommended. Factors to consider include sample preservation; sample storage practices; data use; levels of contamination found in the sample; and the physical, chemical, and biological stability of the target analytes in the sample matrix.

I10 The duplicate sample RPD is greater than the advisory limit and the sample result is a detect. Manual review is suggested to determine the source of the difference between analyses.

I10a The duplicate sample RPD is greater than the advisory limit and the sample result is a nondetect. Manual review is suggested to determine the source of the difference between analyses.

I13a Insufficient sample volume was received for a duplicate-sample analysis.

I13b The duplicate-sample analysis was not performed on a sample associated with this request number.

I14a Insufficient sample volume was received for a matrix-spike analysis.

I14b The matrix-spike analysis was not performed on a sample associated with this request number.

I15 The sample was damaged, lost, or there was insufficient quantity and the analytical laboratory was unable to analyze it.

I15a An ICV was not reported for this sample.

I15b A CCV was not reported for this sample.

Data Validation Cover Sheet

Section I.

Request Number:_____ Validation Date:_____ Lab Code:_____

Contract Laboratory Name:_____

Validator:_____ Organization:_____

Analytical Suite (check all that apply): ☐ Volatile Organics ☐ Semivolatile Organics ☐ Organochlorine Pesticides/Polychlorinated Biphenyls ☐ High Explosives ☐ Inorganics ☐ Radiochemistry

Other (describe): _____

Section II. Completeness Check

Yes	No	n/a	(check one)	Yes	No	n/a	(check one)
☐	☐	☐	1. Chain-of-custody form(s)	☐	☐	☐	6. Raw/BSS data
☐	☐	☐	2. Case narrative	☐	☐	☐	7. Quality control forms
☐	☐	☐	3. Sample result forms	☐	☐	☐	8. Quantitation reports
☐	☐	☐	4. Sample chromatograms	☐	☐	☐	9. TICE forms
☐	☐	☐	5. Standard chromatograms	☐	☐	☐	10. GCs mass spectra

Identify any samples in the assigned Request Number that are missing:

_____ _____ _____ _____ _____ _____ _____ _____

_____ _____ _____ _____ _____ _____ _____ _____

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed upon date of resolution and contract laboratory point of contact):

(Attach additional comment sheets as necessary)

Validator's signature: _____ Date: _____

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Inorganic Data Validation Checklist

Part I. Detection Status Verification

Criterion	Criterion true? (yes or no)	Action if "criterion true?" = yes Assign qualifier & reason code…
Are analytical-laboratory B-flagged results present?	1a.	1b. "**J, I1**" to all affected analytes on the Form I.

Part II. Initial and Continuing Calibrations Verification

Criteria	Criterion true? (yes or no)	Action if "criterion true?" = no Assign qualifier & reason code…	List affected analyte(s) and affected samples.
Is the initial calibration verification (ICV) present?	1a.	1b. "**A, I15a**" for any missing documentation. In block 1c, record all sample matrices and/or analytical batches that did not include an ICV.	1c.
Is the continuing calibration verification (CCV) present?	2a.	2b. "**A, I15b**" for any missing documentation. In block 2c, record all sample matrices and/or analytical batches that did not include a CCV.	2c.
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Part IIIa. Preparation Blank Validation Criteria

Criterion	Criterion true? (yes or no)	Action if "criterion true?" = no Assign qualifier & reason code…	List affected matricies or batches.
Was a preparation blank analyzed for each sample matrix and batch?	1a.	1b. "**A, I4b**" for any missing documentation. In block 1c, record all sample matrices and/or analytical batches that did not include a preparation blank.	1c.

Part IIIb. Preparation Blank Validation Criteria (continued)

Criteria	Criterion true? (yes or no)	Action if "criterion true?" = yes Assign qualifier & reason code…	List detected blank analyte(s) and affected samples.	Analyte concentration (mg/kg)
Is a target analyte detected in the preparation blank **AND** is the same target analyte detected in the sample result < five times the amount detected in the preparation blank **AND** is the target analyte detected in the sample < the EDL?	2a.	2b. "**B, I5**" to affected sample analyte(s).	2c.	2d.

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Part IIIb. Preparation Blank Validation Criteria (continued)

Criteria	Criterion true? (yes or no)	Action if "criterion true?" = yes Assign qualifier & reason code…	List detected blank analyte(s) and affected samples.	Analyte concentration (mg/kg)
Is a target analyte detected in the preparation blank **AND** is the same target analyte detected in the sample result < five times the amount detected in the preparation blank **AND** is the target analyte detected in the sample = the EDL?	3a.	3b. "**U, I4a**" to affected sample analyte(s).	3c.	3d.

Part IVa. ICPES Interference Check Sample (ICS) Validation Criteria

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = no Assign qualifier & reason code…	List all noncompliant ICPES ICS analytes.
Was an ICPES ICS analyzed for each sample matrix and/or analytical batch? ICS samples must contain the following analytes: Ag, Ba, Be, Cd. Co, Cr, Cu, Mn, Ni, Pb, V, and Zn. **Note:** Pb is required only if Pb was analyzed by ICPES.	1a.	1b. "**A, I7d**" for any missing documentation. In block 1c, record • any ICPES ICS analytes not reported and • all affected samples.	1c.

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Part IVb. ICPES Interference Check Sample (ICS) Validation Criteria (continued)

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = yes Assign qualifier & reason code…	List all noncompliant ICPES ICS analytes and samples.
Is ICS analyte recovery > 120%?	2a.	2b. "**J+, I7**" to all detected sample analytes. In block 2c, record • the affected ICPES ICS analytes and • the corresponding samples.	2c.
Is ICS analyte recovery = 50% and < 80%?	3a.	3b. "**J-, I7a**" to all *detected* sample analytes. In block 3c, record • the affected ICPES ICS analytes and • the corresponding samples. "**UJ, I7c**" to all *nondetected* sample analytes. In block 3c, record • the affected ICPES ICS analytes and • the corresponding samples.	3c.
Is ICS recovery <50%?	4a.	4b. "**RPM, I7b**" to all *detected* and *nondetected* sample analytes. In block 4c, record • the affected ICPES ICS analytes and • the corresponding samples.	4c.

Inorganic Data Validation Checklist

Part Va. Matrix-Spike Validation Criteria

Criterion	Criterion true? (yes/no)	Action if "criterion true?" = no Identify noncompliant matrix-spike percent recovery values ***and*** assign qualifier & reason code…	List ER Project RN & sample ID for matrix-spike sample.
Is a matrix-spike sample present?	1a.	1b. "**PM, I14b**" if the matrix-spike sample that was analyzed was on a sample not associated with this request number. In block 1c record • the request number and sample ID of spiked sample. **OR** "**A, I14a**" if the matrix-spike sample was missing because insufficient sample volume was received by the analytical laboratory for the duplicate analysis.	1c.

Inorganic Data Validation Checklist

Part Vb. Matrix-Spike Validation Criteria (continued)

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = yes Assign qualifier & reason code…	List all affected matrix-spike analytes and samples.	Percent recovery
Is the matrix spike percent recovery value > 150%?	2a.	2b. "**J+, I3h**" to all *detected* sample analytes and "**UJ, I3g**" to all *nondetected* sample analytes.	2c.	2d.
Is the matrix spike percent recovery value 125%–150%?	3a.	3b. "**J+, I3**" to all *detected* sample analytes and no qualifier for any *nondetected* sample analytes.	3c.	3d.
Is the matrix spike percent recovery value between 30% and 75%, inclusive?	4a.	4b. "**J-, I3a**" to all *detected* sample analytes and "**UJ, I3e**" to all *nondetected* sample analytes.	4c.	4d.
Is the matrix spike percent recovery value < 30%?	5a.	5b. "**J, I3f**" to all *detected* sample analytes and "**RPM, I3d**" to all *nondetected* sample analytes.	5c.	5d.

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Part Vc. Required Matrix-Spike Compounds and Spiking Levels

Matrix spike name	Soil matrix spiking levels (mg/kg)		Water matrix spiking levels (µg/L)		Water (µg/L) or soil (mg/kg)
	ICPES	GFAA	GFAA	ICPES	Other
Aluminum	—[a]	n/a[b]	n/a	2000	n/a
Antimony	100	20	100	500	n/a
Arsenic	400	8	40	2000	n/a
Barium	400	n/a	n/a	2000	n/a
Beryllium	10	n/a	n/a	50	n/a
Cadmium	10	1	5	50	n/a
Calcium	—	n/a	n/a	—	n/a
Chromium	40	n/a	n/a	200	n/a
Cobalt	100	n/a	n/a	500	n/a
Copper	50	n/a	n/a		n/a
Iron	—	n/a	n/a	1000	n/a
Lead	100	4	20	500	n/a
Magnesium	—	n/a	n/a	—	n/a
Manganese	100	n/a	n/a	500	n/a
Mercury	n/a	n/a	n/a	n/a	1 (CVAA)
Nickel	100	n/a	n/a	500	n/a
Potassium	—	n/a	n/a	—	n/a
Selenium	400	2	10	2000	n/a
Silver	10		n/a	50	n/a
Sodium	—	n/a	n/a	—	n/a
Thallium	400	10	50	2000	n/a
Vanadium	100	n/a	n/a	500	n/a
Zinc	100	n/a	n/a	500	n/a

[a] — = no spike required for this target analyte.
[b] n/a = spike not applicable for this target analyte.

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Part VIa. Duplicate Sample Validation Criteria

Criterion	Criterion true? (yes/no)	Action if "criterion true?" = no Identify noncompliant duplicate percent recovery values *and* assign qualifier & reason code…	List ER Project RN & sample ID for duplicate sample.
Is a duplicate sample present?	1a.	1b. "**PM, I13b**" if the duplicate sample that was analyzed was on a sample not associated with this request number. In block 1c record • the request number and sample ID of duplicate sample. **OR** "**A, I13a**" if the duplicate sample was missing because insufficient sample volume was received by the analytical laboratory for the duplicate analysis.	1c.

Part VIb. Duplicate Sample Validation Criteria (continued)

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = no Assign qualifier & reason code…	List all noncompliant duplicate analytes.	Relative percent difference
Are *both* sample and duplicate-sample results = five times the EDL **AND** the RPDs for *all* analytes = the limit (20% for water samples or = 35% for soil samples) **OR** *either* the sample or duplicate-sample result is < five times the EDL?	2a.	2b. "**J, I10**" to all *detected* sample analytes and "**UJ, I10a**" to all *nondetected* sample analytes. In block 2c record • noncompliant samples and analytes. In block 2d record • the RPDs of the noncompliant analytes.	2c.	2d.

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Part VIc. Duplicate Sample Validation Criteria (continued)

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = yes Assign qualifier & reason code…	List all noncompliant duplicate analytes.	Relative percent difference
Is *either* the sample or duplicate-sample result < five times the EDL **AND** the sample result is *not* within the range of ± 1 times the EDL (± 2 times the EDL for soil samples) of the duplicate-sample result?	3a.	3b. "**J, I10**" to all *detected* sample analytes and "**UJ, I10a**" to all *nondetected* sample analytes. In block 3c record • noncompliant samples and analytes. In block 3d record • the RPDs of the noncompliant analytes.	3c.	3d.

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Part VII. Laboratory Control Sample Validation Criteria

Note: An aqueous LCS must be prepared and analyzed for each aqueous sample request. If a solid LCS is available from a vendor, then a solid LCS may be used for soil samples. If a solid LCS is unavailable an aqueous LCS must be used. Criteria exist for aqueous sample matrix only.

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = yes Assign qualifier & reason code…	List all noncompliant LCS analytes.	LCS %Rs
Was a required LCS *not* associated with this request?	1a.	1b. "**A, I6c**" for any missing documentation. In block 1c, record • any LCS analytes not reported.	1c.	1d. n/a
Is the LCS percent recovery value < 80% for any analyte?	2a.	2b. "**J-, I6a**" to all *detected* sample analytes and "**UJ, I6b**" to all *nondetected* sample analytes. In block 2c record • noncompliant LCS analytes. In block 2d record • the %R values of the noncompliant LCS analytes.	2c.	2d.
Is the LCS percent recovery value > 120% for any analyte?	3a.	3b. "**J+, I6**" to all detected sample analytes. In block 3c record • noncompliant LCS analytes. In block 3d record • the %R values of the noncompliant LCS analytes.	3c.	3d.

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Part VIII. Holding Time Validation Criteria

Note: Holding times are required for water samples associated with mercury and cyanide analyses only. The holding time for mercury water samples is 28 days from sample collection. The holding time for cyanide water samples is 14 days from sample collection.

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = no Assign qualifier & reason code…	List samples for which holding times were exceeded.	List the number of days by which holding times were exceeded.
Was each sample analyzed within its analytical holding time?	1a.	1b. "**PM, I9**" to all analytes in affected samples.	1c.	1d.